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Indian Standard
SPECIFICATION FOR PHENYL-β-NAPHTHYLAMINE

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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April 1986

Indian Standard
SPECIFICATION FOR PHENYL-β-NAPHTHYLAMINE

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Indian Standard
SPECIFICATION FOR PHENYL-β-NAPHTHYLAMINE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 March 1985, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Phenyl-β-naphthylamine is a general purpose heat-resistant and anti-flexcracking antioxidant. It belongs to staining type of antioxidants and is recommended for black or dark coloured rubber goods only. Phenyl-β-naphthylamine is soluble in benzene, sparingly soluble in petroleum ether and insoluble in water. Its solubility in rubber is approximately 1·5 percent.

0.3 Besides specifying chemical requirements, the standard includes a recommended procedure for evaluating the material by means of a standard compounding and vulcanizing procedure.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounding off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for phenyl-β-naphthylamine intended for use as an antioxidant in rubber compounds.

2. REQUIREMENTS

2.1 Description — The material shall be in flake or powder form and light-grey pink in colour.

2.2 The material shall comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 of the table.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR PHENYL-β-NAPHTHYLAMINE

(*Clause* 2.2)

SL No.	CHARACTERISTICS	REQUIREMENTS	METHOD OF TEST, REF TO
(1)	(2)	(3)	(4)
i)	Assay (PBNA content), percent by mass, *Min*	95·0	A-2
ii)	Crystallizing point, *Min* °C	104·0	A-3
iii)	Sulphated ash, percent by mass, *Max*	0·2	A-6 of IS : 6918-1972*
iv)	β-Naphthol, percent by mass, *Max*	0·5	A-4
v)	Volatile amines (as aniline), percent by mass, *Max*	0·1	A-5
vi)	β-Naphthylamine, ppm, *Max*	1·0	A-6
vii)	Copper, ppm, *Max*	20	12 of IS : 7086 (Part 1)-1973†
viii)	Fineness (in case of powder) residue retained on:		3 of IS : 7086 (Part 1)-1973†
	a) 150 micron IS-sieve percent by mass, *Max*	0·1	
	b) 43 micron IS-sieve, percent by mass, *Max*	1·0	

*Specification for mercaptobenzothiazole.

†Methods of sampling and test for rubber compounding ingredients, Part 1.

2.3 Compounding Performance Requirement — The material when compounded and tested as given in Appendix B shall have its properties comparable within the limits prescribed in **B-4.1** with those of the sample complying with the requirements of **2.1** and **2.2** and approved previously by the purchaser.

3. PACKING AND MARKING

3.1 The material shall be packed in 20 or 25 kg packs of multiwalled paper bags or as agreed to between the purchaser and the supplier.

3.2 The packages shall be securely closed and legibly marked with the following information:

a) Manufacturer's name and registered trade-mark, if any;
b) Name of the material;
c) Net mass of the material; and
d) Lot and batch number.

3.2.1 The package may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Sampling — For the purpose of ascertaining the conformity of phenyl-β-naphthylamine in a consignment to the specification, sampling as prescribed in IS : 7086 (Part 1)-1973* shall be followed.

4.2 Number of Tests — Tests for all the characteristics shall be conducted on a composite sample.

4.3 Criteria for Conformity — The lot shall be declared as conforming to this specification if the sample satisfies all the requirements for the various characteristics given in Table 1 and also meets the specified requirements given in Appendix B.

APPENDIX A

(*Table* 1)

TEST METHODS FOR PHENYL-β-NAPHTHYLAMINE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

---

*Methods of sampling and test for rubber compounding ingredients, Part 1.

†Specification for water for general laboratory use (*second revision*).

A-2. ASSAY (DETERMINATION OF PBNA CONTENT)

A-2.1 Outline of the Method

A-2.1.1 Nitrogen content of phenyl-β-naphthylamine is determined and the PBNA content is calculated by using the following formula:

$$\text{PBNA content percent by mass} = \text{Percent nitrogen content in PBNA} \times \frac{219{\cdot}3}{14{\cdot}01}$$

A-2.2 Determination of Nitrogen Content — The nitrogen content in PBNA can be conveniently determined by Kjeldahl method in which the nitrogen is converted into ammonium sulphate by oxidation. The ammonium sulphate is subsequently decomposed by boiling with sodium hydroxide solution. The evolved ammonia is distilled off, absorbed in a solution of boric acid and titrated with standard acid in presence of a suitable indicator.

A-2.2.1 *Apparatus*

A-2.2.1.1 Kjeldahl flask, 300 ml capacity.

A-2.2.1.2 Distillation unit as shown in Fig. 1.

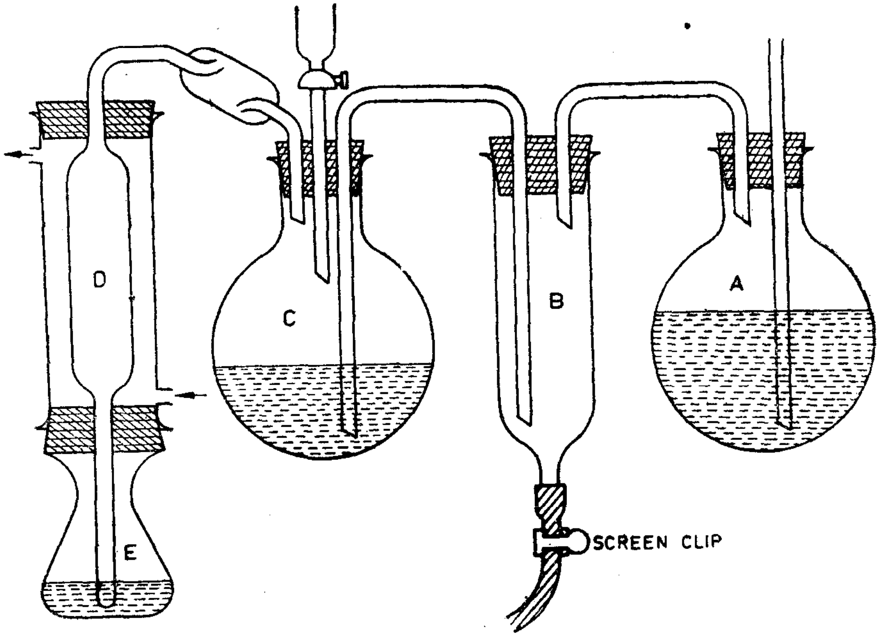

FIG. 1 DISTILLATION UNIT

A-2.2.2 *Reagents*

A-2.2.2.1 *Catalyst mixture* — Carefully grind together 300 g of potassium sulphate and 12 g of mercury oxide in a clean dry mortar. Mix well by rotating on a roller mill for 1 hour.

A-2.2.2.2 *Boric acid solution* — Dissolve 40 g of boric acid (AR/GR grade) in water, warming if necessary, and make up the volume to 2 litres. Add 20 ml methanol red solution (0·05 percent) and 0·4 ml of methylene blue solution (1·5 percent) and mix well. When one drop of N/10 sodium hydroxide is added to 20 ml of the solution, the colour shall entirely change from purple to green. The solution can be kept for at least three months without deterioration.

A-2.2.3 *Procedure* — Weigh accurately 1 g sample of PBNA with 0·0001 g accuracy and transfer it into 300 ml Kjeldahl flask, add 10 to 12 g catalyst mixture and 20 ml concentrated sulphuric acid (SG 1·85). Heat to boil on a digestion bench until the mixture becomes clear and almost colourless. It is important that heating should be slow initially to avoid foaming (if any) followed by vigorous boiling to cause sulphuric acid to reflux 1/3 of the way up to the neck of Kjeldahl flask. Boil for one hour.

A-2.2.3.1 Set up the distillation assembly as shown in the Fig. 1. Take 50 ml of boric acid solution into flask *E* and place the flask below the condenser in such a way that the end of the inner tube of the condenser is below the surface of the solution. The flask *E* should be kept in a ice cold water. Add a piece of porous pot/granulated zinc to the distillation flask *C*. Cool the Kjeldahl flask and its contents and transfer the acid quantitatively into flask *C* through the funnel. Kjeldahl flask should be rinsed with distilled water three times with 100 to 150 ml water in all and the contents should be transferred to flask *C*. Add few drops of methyl red indicator through the funnel followed by 7·5 M sodium hydroxide slowly until the mixture is just alkaline. Add further approximately 20 ml of 7·5 M sodium hydroxide. In the meantime, generate steam in *A* keeping the screw clip in open until *B* is full of steam and then close screw clip in *B*. Steam distill until about 200 ml of distillate has been collected. It is important that only tip of the condenser's inner tube is dipping into the boric acid solution to avoid any back suction. Remove flask *E* and wash the inner tube of condenser with distilled water and transfer the content to flask *E*. Titrate the solution in *E* with N/10 hydrochloric acid. Carry out blank also under identical conditions without sample.

Calculate the percentage nitrogen from this formula:

$$\text{Percent nitrogen} = \frac{V_2 - V_1 \times N \times 0{\cdot}01401 \times 100}{M}$$

where

V_2 = volume in ml of standard acid used in titration,

V_1 = volume in ml of standard acid used in blank titration,
N = normality of acid, and
M = mass in g of the sample.

A-3. DETERMINATION OF CRYSTALLIZING POINT

A-3.1 The crystallizing point of an organic compound is often a good criterion of the purity of the material. Its determination is in general to be preferred to a melting point determination.

A-3.1.1 This method, however, has several limitations. It cannot be easily applied to materials which will melt at a temperature above 140°C. It obviously cannot be applied to compounds which decompose or alter on melting. This method is frequently used in determination of one isomer in the presence of other isomers.

A-3.2 Apparatus — A boiling tube approximately 150 × 25 mm is placed inside a larger tube 180 × 38 mm which acts as an air jacket and which is weighed with lead shots. The inner tube is closed by a cork which carries a suitable thermometer having at least count of 0·1°C or 0·2°C in the centre and also a thin looped metal/glass stirrer. The outer tube is suspended in a water bath or other medium, the temperature of which is so adjusted that at the beginning of the test it is 5°C below the expected crystallizing point of the test material. At the end of the test, the temperature should not be more than 10°C below the crystallizing point.

NOTE 1 — The thermometer used in the determination should be immersed to a depth of 10 cm in the molten material. In the case of thermometer calibrated for 10 cm immersion the observed reading is taken as correct and a correction for emergent stem is not necessary. In the case of a thermometer calibrated for total immersion a correction for emergent stem must be applied.

The corrected temperature reading is obtained from the formula:

$$T = T_0 + 0{\cdot}00143\ (T_0 - t) \times N$$

where

T_0 = observed temperature,
t = mean temperature of emergent stem, and
N = number of degrees on emergent stem.

If there is insufficient material to allow 10 cm immersion, a correction for emergent stem should be applied although this will be only approximate. In order to keep the correction for emergent stem as low as possible a short range thermometer shall be used and a thermometer which requires a correction for emergent stem greater than 0·5°C shall not be used.

NOTE 2 — The bottom of the bulb of the thermometer shall not be less than 1 cm from the bottom of the crystallizing point tube.

NOTE 3 — In repeat determinations of crystallizing point, a fresh portion of the sample shall be taken. On no account the material from one test be remelted and used for the duplicate test.

A-3.2.1 *Preparation of the Sample for the Test* — Most substances for which crystallizing point is required, are tested as it is. Certain materials, however, contain water in varying proportions. These have to be subjected to preliminary drying treatment before determination of crystallizing point. Method for predrying could be selected depending upon the material to be tested.

A-3.3 **Procedure** — Fill the inner tube to approximately 2/3 of its capacity with the solid and immerse in a bath of suitable liquid at a temperature about 10°C above the expected crystallizing point. Withdraw the tube from the bath when all the material gets melted and its temperature is 5°C to 10°C above the expected crystallizing point. Wipe off adherent liquid from the outer side of the tube quickly with a wad of dry cotton, wool or tissue paper and put it in the outer cooling packet. Allow the contents of the tube to cool with continuous but gentle stirring. Take readings of the thermometer at regular intervals of 30 seconds. If the temperature falls and then remains constant for five consecutive readings, this constant temperature is taken as the crystallizing point.

If super cooling takes place, observe constant temperature after the temperature rise. A temperature rise of 1°C is to be regarded as maximum allowable.

A-4. DETERMINATION OF β-NAPHTHOL

A-4.1 Procedure

A-4.1.1 Weigh accurately 20 g of prepared sample with a 0·000 1 g accuracy into a 500 ml conical flask, add 250 ml of toluene and dissolve by warming in a water bath. Cool and transfer the contents to a 500 ml separating funnel. Shake with 100 ml normal sodium hydroxide solution, allow to settle and take off the aqueous layer into one litre beaker. Repeat the extraction of the toluene layer with 50 ml normal sodium hydroxide as mentioned above followed by two washings separately with 50 ml water and collect all the alkaline and aqueous washings into one litre beakers.

A-4.1.2 Neutralize the combined alkaline and aqueous washings with 3 molar acetic acid, add 5 g sodium acetate crystals followed by few pieces of washed ice and titrate with N/20 *p*-nitro diazobenzene till a drop of the diazo solution run into a clear rim round a drop of the titrated solution spotted on a filter paper just fails to give a colour.

Calculate the percentage of β-naphthol as per formula given below:

$$\text{Percent } \beta\text{-naphthol} = \frac{B \times 0{\cdot}72}{M}$$

where

B = volume of N/20 *p*-nitro diazobenzene solution used for titration, and

M = mass of sample taken.

A-5. DETERMINATION OF VOLATILE AMINES

A-5.1 Procedure

A-5.1.1 Weigh accurately 10 g prepared sample, add 100 ml water and steam distil, collecting the distillate in 100 ml portions. Cool each portion to room temperature, filter if necessary, add 10 ml of 20 percent m/v sodium bromide solution followed by 10 ml concentrated hydrochloric acid (specific gravity 1·18).

Titrate at 25°C with 0·2 N potassium bromate until an immediate blue colour is obtained on spotting a drop of the titrated solution on starch iodide paper 2 minutes after the last addition of bromate solution added 0·1 ml at a time towards the end of the titration. Continue distillation until two consecutive 100 ml. portions treated as above give the sane titration (which must be less than 1 ml). Treat the final titration as a blank titrate and deduct its value separately from all the titrations made.

Calculate the percent volatile amines as per formula given below:

Percent volatile amine (calculated at aniline)

$$= \frac{A \times 0{\cdot}31}{M}$$

where

A = total volume of 0·2 N potassium bromate solution used, and

M = mass of sample taken.

A-6. DETERMINATION OF β-NAPHTHYLAMINE

A-6.1 Method

A-6.1.1 *Outline of the Method* — β-naphthylamine is extracted from a toluene solution of the sample into dilute hydrochloric acid. The aqueous phase is made alkaline to nitrazine yellow and extracted with toluene. The β-naphthylamine content of this solution is determined by gas liquid chromatography.

A-6.1.2 *Procedure* — Carry out the test under the following conditions using an ISO thermal gas chromatograph fitted with flame ionization detector.

a) Column — 1 meter long packed with acid washed celite (100 — 120 mesh/potassium hydroxide/carbowax 20 M ratio 100 : 5 : 2) injection region packed with above packing.

b) Temperature-column and injection region 190°C.

c) Carrier gas flow rate nitrogen, 60 ml/min.

d) Attenuator control position — Adjust the controls to give a chart similar to Fig. 2.

e) Chart speed 50 cm/hour.

A-6.1.3 *Preparation of Standard and Sample*

a) *Standard* — Weigh accurately 0·01 g (up to 0·000 1 g) pure β-naphthylamine into a 100 ml volumetric flask. Dissolve it with toluene (chromatographic grade) and make up the volume up to the mark. Mix well. Call this solution as A. Take 5 ml of aliquot from solution A into a 50 ml volumetric flask and make up the volume to 50 ml mark with toluene and mark it solution B.

b) *Sample* — Dissolve 25 g of the prepared sample in 250 ml beaker and dissolve it with 150 ml of toluene (chromatographic grade) warm the solution, if necessary. Cool and transfer the contents to 500 ml separating funnel. Extract with 2 × 30 ml portions of 1 normal hydrochloric acid. Combine the acid extracts, make it alkaline with 10 percent m/v solution of sodium carbonate to nitrazine yellow colour. Extract with 5 ml toluene. Separate and transfer the toluene layer to a vial. Call the solution C.

c) Load 5 μ of solutions B and C.

A-6.1.4 *Method of Assessing Results* — Compare the record obtained from standard and test sample, measure the areas under β-naphthylamine peak for both and calculate β-naphthylamine as under.

β-naphthylamine (ppm)

$$= \frac{\text{Area of (β-naphthylamine peak) in solution C}}{\text{Area of (β-naphthylamine peak) in solution B}} \times \frac{\text{mass in g of β-naphthylamine in Solution A}}{\text{mass in g of sample taken}} \times 5\,000$$

NOTE — Using the mass given above, the standard is equivalent to 2 ppm β-naphthylamine.

Caution — Since β-naphthylamine is hazardous to health, a great care must be taken during preparation and handling of its solutions to avoid contact with skin. As a precautionary measure, it is recommended to use gloves while working. Any solid or solution coming in contact with the skin must be washed off instantaneously with soap and water. In no case β-naphthylamine solution should be pipetted.

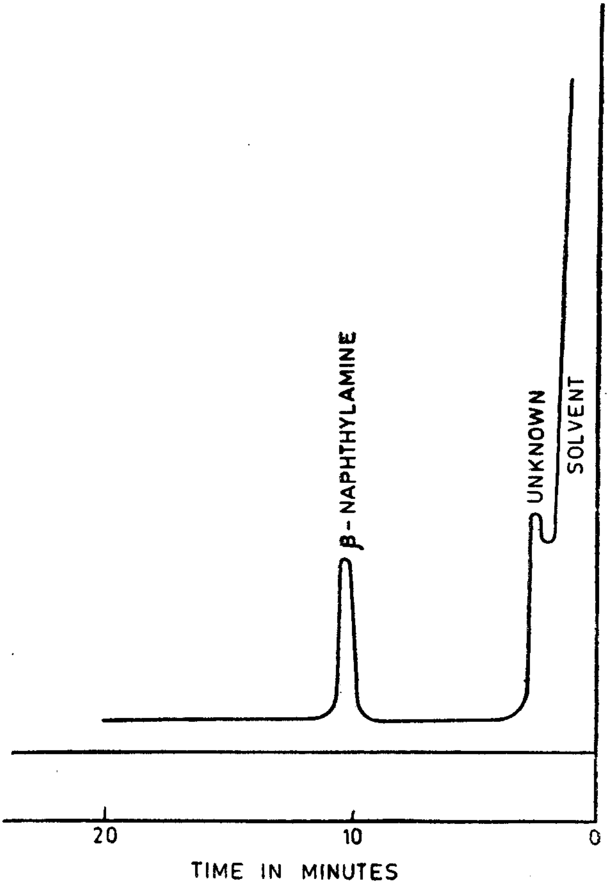

Fig. 2 Representative Curve of Gas Liquid Chromatograph

APPENDIX B

(*Clauses* 2.3 *and* 4.3)

METHOD FOR COMPOUNDING AND TESTING PHENYL-β-NAPHTHYLAMINE

B-1. TEST COMPOUND

B-1.1 For guidance, the test compound with the following composition may be used for testing performance properties of phenyl-β-naphthylamine in rubber compound:

	Part by Mass
Natural rubber grade IS NR 5 (*see* IS : 4588-1977*)	100
Zinc oxide (*see* IS : 3399-1973†)	3·5
Stearic acid (*see* IS : 1675-1971‡)	3·0
Rubber process oil (Naphthenic type)	3·5
HAF carbon black (*see* IS : 7497-1974§)	50·5
Sulphur (*see* IS : 8851-1978‖)	2·5
Benzothiazyl-2-cyclohexyl sulphanamide (*see* IS : 7069-1973¶)	0·5
Phenyl-β-naphthylamine	1·0

B-2. COMPOUND

B-2.1 Follow the procedure prescribed in NR : 9 of IS : 3660 (Part 1)-1972**.

B-3. TESTS

B-3.1 The tests given below are recommended for each test sample. The approved sample shall also be tested in the same compound side by side.

---

*Specification for rubber, raw, natural (*second revision*).

†Specification for zinc oxide for rubber industry (*first revision*).

‡Specification for stearic acids, technical (*first revision*).

§Specification for high abrasion furnace (HAF) carbon black.

‖Specification for sulphur for rubber industry.

¶Specification for benzothiazyl-2-cyclohexyl sulphanamide.

**Methods of test for natural rubber, Part 1 (*first revision*).

B-3.1.1 Tensile strength, modulus of 300 percent elongation and elongation at break at different cures at 141°C stepwise, from below to above the expected optimum cure, shall be determined to ascertain the optimum cure time.

Tensile properties (that is tensile strength and elongation at break) of the vulcanisates cured at the ascertained optimum cure time are determined both before and after heat ageing at 70°C for 2, 4 and 6 days.

B-4. RESULTS

B-4.1 The tensile strength and elongation at break, both of original sample and the aged samples shall be within ±10 percent of the values obtained with the approved sample tested at the same time.

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RUBBER CHEMICALS

IS :	
917-1976	Activated calcium carbonate for rubber industry (*first revision*)
1683-1973	Barytes for rubber industry (*first revision*)
1684-1973	Natural red oxides of iron for rubber industry (*first revision*)
1685-1975	Whiting for rubber industry (*first revision*)
3399-1973	Zinc oxide for rubber industry (*first revision*)
6918-1972	Mercapto-benzothiazole
7069-1973	Benzothiazyl-2- cyclohexyl sulphanamide
7086 (Part I)-1973	Methods of sampling and test for rubber compounding ingredients, Part I
7351-1974	Styrenated phenol
7497-1974	High abrasion furnace (HAF) carbon black
7498-1974	Methods of sampling and test for carbon black
8134-1976	Intermediate super abrasion furnace (ISAF) carbon black
8135-1976	Fast extrusion furnace (FEF) carbon black
8219-1976	Synthetic red oxide of iron for rubber industry (excluding venetian red)
8483-1976	Dibenzothiazyl disulphide
8851-1978	Sulphur for rubber industry
8862-1978	Titanium dioxide (Anatase type) for rubber industry
8891-1978	Pine tar for rubber industry
8979-1978	Tetramethyl thiuram disulphide
9406-1980	Calcium silicate for rubber industry
9407-1980	Light magnesium oxide for rubber industry
10130-1982	Vulcanized vegetable oils (factice) for rubber industry
10357-1982	General purpose furnace (GPF) carbon black
10358-1982	Super abrasion furnace (SAF) carbon black
10387-1982	Semi-reinforcing furnace (SRF) carbon black.